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USE OF THE INTERFEROMETER IN GAS ANALYSIS

BY

FRANK M. SEIBERT AND WALTER C. HARPSTER

ENGINEERING

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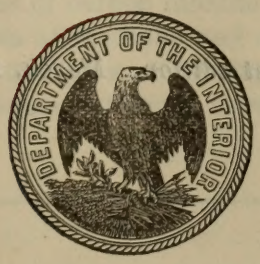

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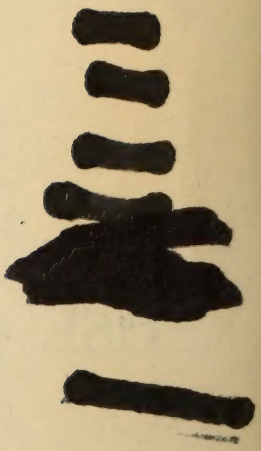

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USE OF THE INTERFEROMETER IN GAS ANALYSIS.

By Frank M. Seibert and Walter C. Harpster.

INTRODUCTION.

This paper describes the outcome of some of the investigations made by the Bureau of Mines in connection with work on mine gases and natural gas. For special work in the field of gas analysis some of the older analytical methods have been partly supplanted by those that permit analyses at frequent intervals. The Rayleigh interferometer, as adapted to gas analysis by Fritz Haber and Frederick Lowe, of Jena, meets this requirement. Mohr,[a] Haber,[b] Lowe,[c] and Kuppers,[d] have published studies on the use of the interferometer in gas analysis.

This instrument, which makes use of the optical properties of gases, is suitable for analyzing a binary gas mixture.

TWO TYPES OF INTERFEROMETER.

The interferometer is made in two different models. A laboratory type (Pl. I, *A* and *B*), which is described in this paper, has gas chambers 1 meter long and is employed for exact gas analysis; its precision is equivalent to that of ordinary volumetric gas-analysis apparatus which use mercury as the confining fluid and give results accurate within 0.02 to 0.03 per cent.

The other model (Pl. I, *C*), a short or portable type, is employed for analysis of gas mixtures when less accuracy is desired. Its accuracy is comparable with that usually obtained in technical gas analysis when water is used as the confining fluid, that is, 0.20 to 0.30 per cent of carbon dioxide or methane. In both instruments the principle is the same, the smaller type differing in details essential to portability. The short type when in position is upright; it is about 10 cm. in diameter and 50 cm. high, and weighs about 11 pounds. Its use, like that of the laboratory type, is confined to binary gas mixtures.

DESCRIPTION OF THE LABORATORY TYPE.

Figure 1 is a diagrammatic sketch of the laboratory instrument. The pencil of parallel rays that comes from the light of a Nernst

[a] Mohr, O., Die Verwendung des Zeisschen Interferometers zur technischen Rauchgasanalyse: Ztschr. angew. Chem., Jahrg. 25, June 28, 1912, pp. 1313, 1317.

[b] Haber, F., Optische analyse der Industriegase: Ztschr. angew. Chem., Jahrg. 19, Aug. 17, 1906, pp. 1418–1422.

[c] Lowe, F., A new interferometer for gases and liquids: Physikalische Ztschr., Jahrg. 11, 1910, pp. 1047–1051.

[d] Kuppers, E., Die Bestimmung des Methangehaltes der Wetterproben mit Hilfe des tragbaren Interferometers: Glückauf, Jahrg. 49, Jan. 11, 1913, pp. 47–50.

lamp and passes through the object glass of a collimator *d* travels in three parts to the observation telescope *h*, where it produces an image of the slit, with Frauenhofer diffraction bands, which appear as two straight parallel dark bands in the white field with colored bands on both sides. The upper half of the pencil of rays in the form of a half cylinder passes directly toward a double diaphragm at the end of, and completely covering, the object glass of the telescope. Plate *l* serves only to deflect this pencil downward and blot from the field the dark band caused by the thickness of the metal tubes that confine the gases. This pencil of rays passes through the diaphragm to the upper half of the object glass of the telescope.

The lower half of the pencil of rays is divided into two equal parts by the metallic partition *a b* between the gas chamber *c* and the chamber *e* for the gas used for comparison. Each point of the pencil of rays also passes through a compensator plate *f* and *g*. The part

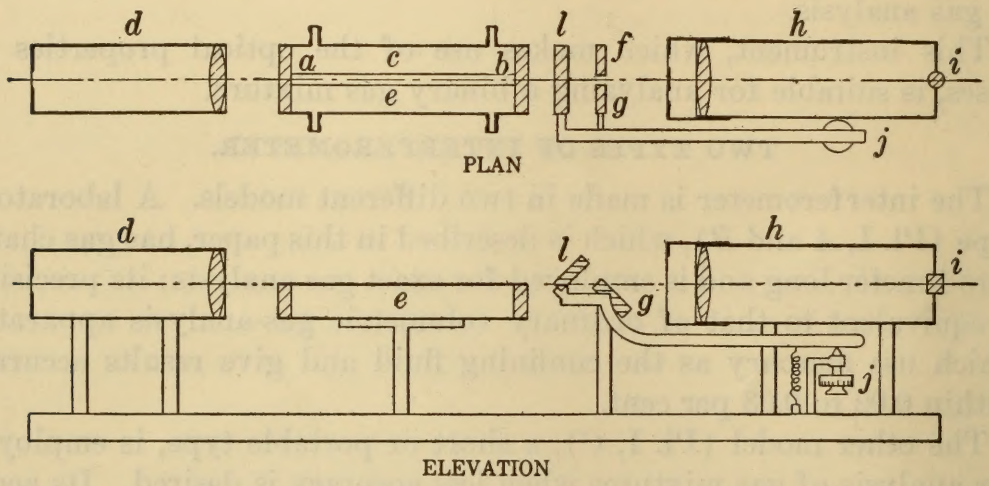


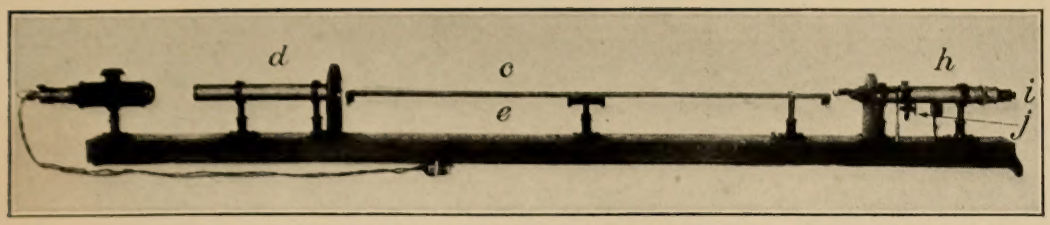
FIGURE 1.—Plan and elevation of laboratory type of interferometer.

g can be moved by a micrometer screw that serves to measure the deflection of the plate; the part *f* is fixed. Thus the optical path of the gas in the chamber *e* can be varied at will. The rays then pass through the diaphragm into the telescope, which is shown in Plate I, *B*.

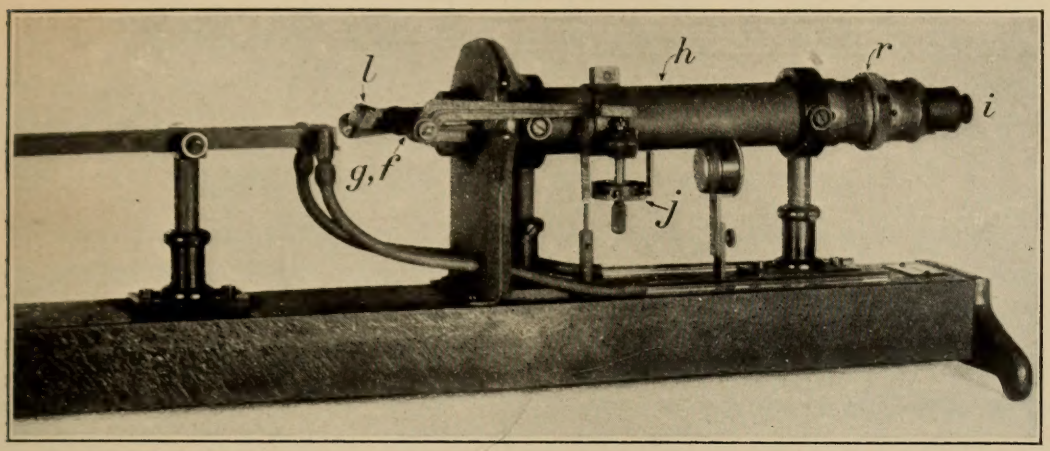
The effects produced by the pencil of rays passing through the system must be viewed by a strongly magnifying cylindrical eyepiece that magnifies only in one direction, as the dark interference bands are only a few hundredths of a millimeter apart.

The band image of the upper half of the rays remains constant during all the manipulation of the apparatus; it corresponds in a way to a cross hair.

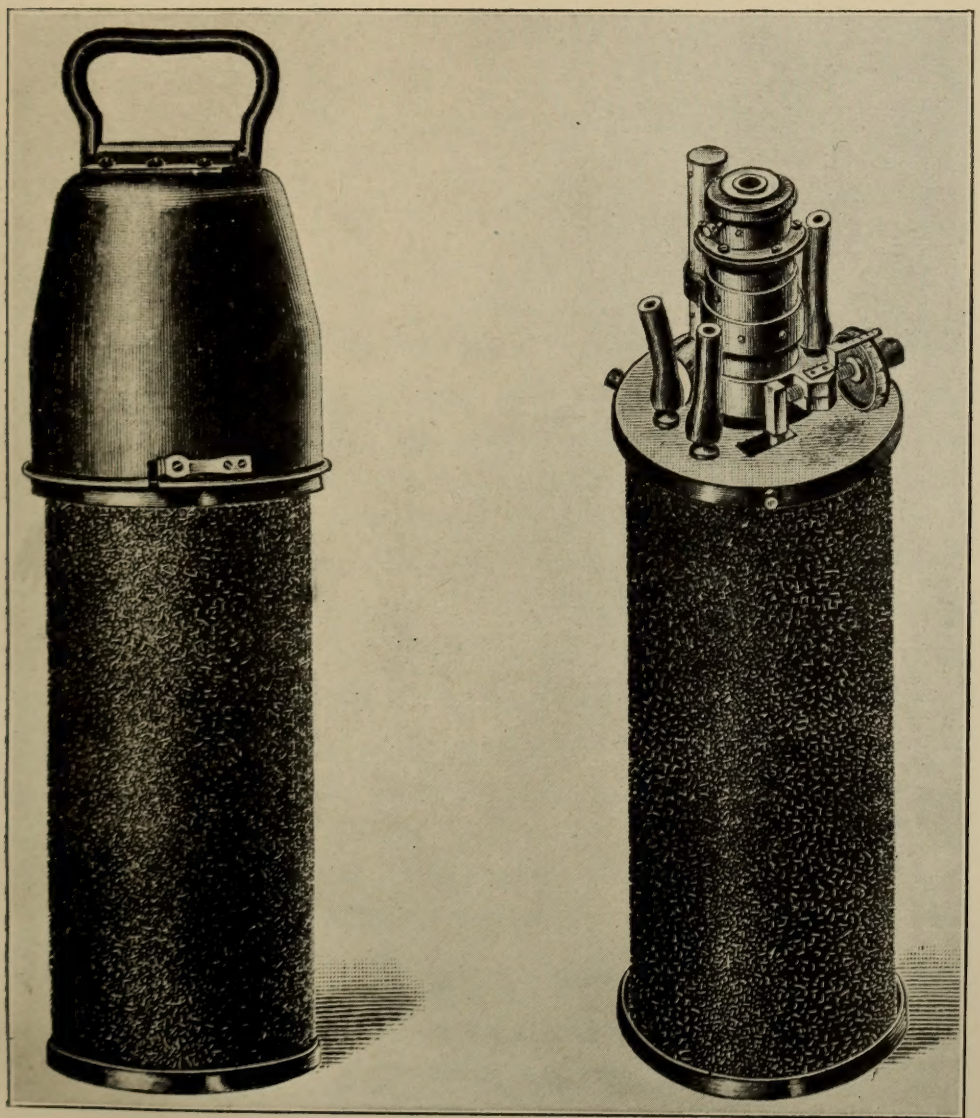
When the two chambers *c* and *e* (fig. 1) are filled with the same gas at the same temperature and pressure and the compensator plates are perfectly parallel, the interference effects produced by the lower half of the pencil of rays can not be distinguished from those produced by the upper half.



A. LABORATORY TYPE OF INTERFEROMETER.



B. TELESCOPE OF INTERFEROMETER; LETTERING OF PARTS CORRESPONDS TO THAT IN FIGURE 2.



C. PORTABLE TYPE OF INTERFEROMETER.

If, however, some other gas, say a mixture of carbon dioxide and air, is introduced into the chamber *c* (fig. 1) at the same temperature and pressure as the gas in *e* (fig. 1), then the lower band image is shifted laterally. By manipulating the screw *j* (Pl. I, *B*) these bands are brought back to their old or zero position. The difference of the readings on the compensator before and after introducing the gas mixture is a measure of the refractive difference of the two gases. The gases in the two chambers must have the same temperature and pressure and must be dry.

PRODUCTION OF INTERFERENCE BANDS.

The manner in which interference bands are produced is important. The explanation of the theory by Young[a] is as follows: The source of light, or the slit of the collimator, is at *i* (fig. 2). The direction of the vibrations sent out from *a* and *b* to any point, *p*, of a screen will conspire when they arrive at *p* in the same phase, and the

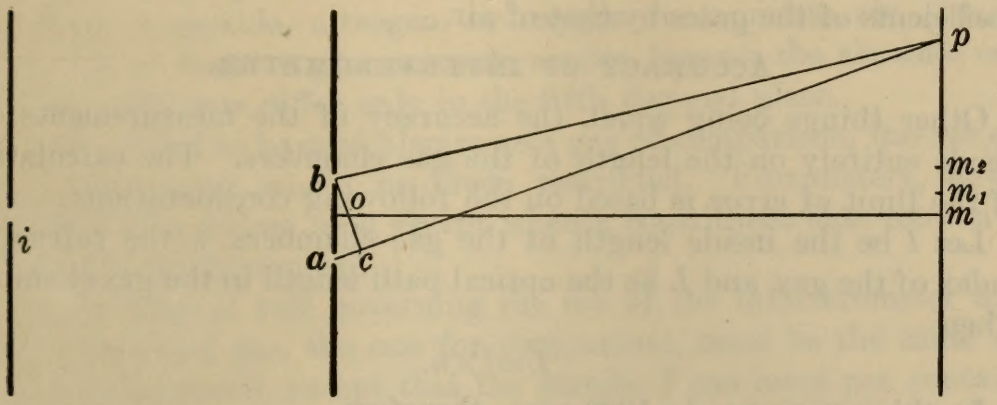


FIGURE 2.—Diamgrammatic representation of production of interference bands.

amplitude of the disturbance will be doubled. If the waves start from *a* and *b* in the same phase they will arrive at *p* in the same phase, provided the path *a p* is equal to the path *b p* or differs from it by any number of complete wave lengths. But the vibrations will be opposed at *p* if the waves arrive there in opposite phases; that is, if *a p* differs from *b p* by one-half wave length or any odd number of half wave lengths. If λ represents the wave length, then when $ap-bp=n\frac{\lambda}{2}$ the point *p* on the screen will be very bright. At m_1 there will be darkness if the difference of its distances from *a* and *b* is half a wave length. At m_2 there will be brightness again if the difference of its distances is two half wave lengths. Similarly there will be darkness again at m_3, brightness at m_4, and so on.

As the differences of the distances of any point m_n from *a* and *b* is $\frac{n\Delta,}{2}$ the locus of m_n in the plane of the paper is an hyperbola, in space it is an hyperboloid.

[a] Preston, Thomas, Theory of light, 4th ed., 1912, pp. 146–149.

As the distance of any fringe from the center depends on the wave length, the use of composite light produces rainbow-colored in place of light and dark bands. The inner edge of these colored bands is violet and the outer, which represents longer wave lengths, is red.

By superposing the two sets of interference bands and by making one-half of the lower set pass through a medium of different optical density the superposition may be destroyed and compensated for at will.

The following values for the refractivities of the principal gases are given by Ramsay and Travers:[a]

Refractivities of gases.

Air=1.00.	Nitrogen=1.016.	Helium=0.1238.
Hydrogen=0.473.	Neon=0.2345.	Krypton=1.449.
Oxygen=0.924.	Argon=0.968	Xenon=2.364.

These values are obtained by dividing the absolute refraction coefficients of the gases by that of air.

ACCURACY OF INTERFEROMETER.

Other things being equal, the accuracy of the measurements depends entirely on the length of the gas chambers. The calculation of the limit of error is based on the following consideration:

Let l be the inside length of the gas chambers, n the refractive index of the gas, and L be the optical path length in the gas chamber. Then

$$L=l\times n.$$

In this apparatus l=1000 mm., therefore

$$L=1000\ n.$$

If n=a small change in the refractive index, then

$$L=l\times n.$$

If the path difference of 1/20 of a wave length be assumed in adjusting the screw when λ=1 wave length and λ=600 mm., then

$$\frac{600}{20}=1000\text{ mm.}\times n.$$

$$n=\frac{30}{1{,}000}=\pm 0.000{,}000{,}03.$$

Evidently the refractive index must be measured with great precision for accurate work.

As the shifting of the bands depends on the difference between the refraction coefficients of the gases in the tubes, the drum is divided so that one division=3×10^{-8}.

If S equals the number of drum divisions that bring the bands back to the zero position, then, $S=1/\times 1\text{x}\times 10^{-8}$, and one is enabled to as-

[a] Ramsay, William, Travers, M. W., On the refractivities of air, oxygen, nitrogen, argon, hydrogen, and helium: Proc. Roy. Soc., vol. 62, 1898, p. 228. On argon and its companions: Phil. Trans. Roy. Soc., vol. 197A, 1901, p. 81.

certain the number of divisions S that corresponds to the difference in composition of the gases in the tubes. This difference can be obtained from a table of the refraction exponents. However, the refraction exponents are imperfectly known and they vary inversely as the absolute temperature and directly as the pressure.

The percentage error made in determining one component of a mixture depends also on the choice of the gas of comparison, and the error can be represented by a formula.

It is better, therefore, to calibrate the instrument empirically, that is, by comparing the indications of the interferometer with the determinations made by some exact analytical method. This was done by the authors. In order to obtain an accuracy of about 0.02 per cent when using as a gas of comparison the gas that predominates in the mixture, the absolute refraction coefficients should differ by about 0.0001, that is, one in the fourth decimal place.

Therefore the greater the difference of the coefficients of absolute refraction the greater is the accuracy of the method. Consequently, if carbon monoxide, nitrogen, or oxygen contaminate air, the interferometer does not give accurate results, because the absolute refraction coefficients differ only in the fifth decimal place.

If air had to be used always as a gas of comparison, the use of the interferometer would be much restricted. Fortunately, however, pure air is seldom used except when a constituent that contaminates air is to be determined.

The general rule governing the use of the interferometer is, that the standard gas, the one for comparison, must be the same as the gas to be tested, except that the standard gas must not contain the constituent to be determined, this constituent having been removed by some suitable means. Then the difference in refraction is caused solely by the constituent to be determined.

METHOD OF CONDUCTING A TEST.

To establish the zero point, air may be aspirated through the drying and soda-lime tubes of the apparatus until the reading is constant; this reading may be used as the zero, or the screw may be set at zero and the "fixed" plate moved by gentle taps exactly into position. The gases passed into the chambers must have the same temperature, pressure, and moisture content. A small wash bottle of concentrated sulphuric acid serves as a drier and also indicates the rate of flow. A phosphorous pentoxide tube may be added to remove the last traces of moisture. For obvious reasons the total volume of this system should be as small as possible. The authors found that about 200 or 300 c.c. must be passed through before the reading became constant.

The opposite ends of the chambers are fitted with capillary tubes to prevent diffusion and insure equality of pressure.

Figure 1 shows that all parts are mounted on a single optical axis; hence careful lateral adjustment is necessary. If the adjustment is disturbed its correction is a matter of considerable difficulty. The lens in front of the Nernst lamp should be so adjusted that the slit in the collimator is in its focus. Different intensities of the two black bands in the center of the field indicate faulty adjustment of the light.

The ability to take readings is acquired quickly; it has been found that the average observer makes adjustments to about one-twentieth of the width of the black bands. Complete mastery of the instrument comes from considerable experience.

RESULTS OF EXPERIMENTS.

Tables 1 and 2 following present the determinations of carbon dioxide and methane mixed with air. The results are for both interferometric and eudiometric analysis, and by the latter are accurate to within 0.02 per cent.

In making these tests with the interferometer the standard gas was air free from carbon dioxide and moisture. At each determination the flow of the gas to be tested was interrupted and the pressure of the gas was brought to that of the atmosphere. When readings were made promptly the diffusion of outside air into the gas chambers was inappreciable.

TABLE 1.—*Analyses of mixtures of carbon dioxide and air.*

CO_2 by eudiometric analysis. A.	CO_2 by interferometer. B.[a]	Column A. / Column B. C.[b]
Per cent.	*Per cent,*	
0.76–0.79	0.77	0.0100
.80– .84	.79	.0104
1.63–1.60	1.64	.00985
2.52–2.55	2.56	.00990
3.67–3.68	3.65	.01007
.26– .28	.28	.00982
.11– .13	.12	.01000
.10– .12	.09	.01222
4.72–4.73	4.77	.00990
8.78–8.81	8.76	.01004

[a] Figures in this column represent difference, in drum divisions, between initial and final readings.
[b] Figures in this column represent the value of 1 division of the drum in terms of carbon dioxide percentage.

TABLE 2.—*Analyses of mixtures of methane and air.*

Methane by eudiometric analysis. A.	CO_2 by interferometer. B.[a]	Column A. / Column B. C.[b]
Per cent.	*Per cent.*	
0.04	0.03	0.01333
.12–0.12	.12	.01000
.41– .41	.42	.00976
.86– .89	.84	.0142
1.53–1.55	1.54	.01000

[a] Figures in this column represent difference, in drum divisions, between initial and final readings.
[b] Figures in this column represent the value of 1 drum division in terms of methane percentage.

EFFECT OF LOWERING OXYGEN CONTENT OF MIXTURES ANALYZED.

As some mixtures do not contain oxygen and nitrogen in the same proportions as air, experiments were made to ascertain the effect of less oxygen and more nitrogen than air contains. The results are given in Table 3 following.

TABLE 3.—*Mixtures of carbon dioxide and air with lowered oxygen content.*

Carbon dioxide by interferometer.	Carbon dioxide by eudiometric analysis.	Oxygen by analysis.	Difference of oxygen content from normal air.	Column 1—column 2 / Column 4.
1	2	3	4	5
Per cent.	*Per cent.*	*Per cent.*		
0.20	0.20	20.77	0.16	.....
.50	.40	20.19	.74	0.135
.71	.63	20.15	.78	.103
.89	.76	19.92	1.01	.128
.57	.43	20.03	.90	.155
.53	.25	19.20	1.73	.162
1.36	1.10	19.31	1.62	.160

The results show that over the range investigated for each 1 per cent decrease of oxygen and corresponding increase of nitrogen the carbon dioxide determined by the interferometer will appear about 0.15 per cent too great if normal air be used as the gas of comparison.

Results of similar experiments with mixtures of methane and air are given in Table 4.

TABLE 4.—*Mixtures of methane and air with lowered oxygen content.*

Methane by interferometer.	Methane by eudiometric analysis.	Oxygen by analysis.	Difference of oxygen per cent from normal air.	Column 1—column 2 / Column 4.
1	2	3	4	5
Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	
0.11	0.08	20.53	0.40	.....
.18	.17	20.36	.57	.....
.84	.66	19.90	1.03	0.174
.44	.16	19.36	1.57	.179
.42	.20	19.33	1.60	.138
.48	.21	19.13	1.80	.150
1.58	1.18	18.53	2.40	.167
.95	.46	18.20	2.73	.179

The results show that over the range investigated for each 1 per cent decrease of oxygen and corresponding increase of nitrogen the methane content indicated by the interferometer will appear about 0.16 per cent too great if normal air be used as the gas of comparison.

CALIBRATION OF INTERFEROMETER FOR OXYGEN-NITROGEN MIXTURES.

In analyzing oxygen obtained from liquid air a rapid method of determining the percentage of oxygen and of nitrogen is necessary. As liquid air evaporates, the residue becomes richer in oxygen. If a part of the distillate be led through the interferometer the variations in composition can be ascertained at all times.

The manner of constructing a calibration curve was as follows: The standard gas used was pure oxygen. It is important that this standard oxygen be pure, especially for investigating the narrow part of the curve. The oxygen from ozone generators was found unsatisfactory, and the oxygen used was obtained by heating pure potassium permanganate; this decomposes according to the equation $2KMnO_4=K_2MnO_4+MnO_2+O_2$.

The oxygen was collected in an aspirator bottle, due precaution being taken against contamination by other gases, and was passed slowly into the chamber *e* (fig. 1) through a small wash bottle of concentrated sulphuric acid, a small soda-lime tube, and a phosphorus pentoxide tube. The other end of the chamber was connected with the atmosphere through a capillary tube 6 inches long. After the air in the system had been completely displaced, one or two bubbles per second were sufficient to prevent diffusion of the air into the chamber through the capillary tube.

After the standard oxygen had been prepared, pure nitrogen was passed through the other tube (through a similar train of sulphuric acid, soda-lime, and phosphorus pentoxide tubes) to obtain the extreme readings of the two gases. Analyzed mixtures of the two gases were then introduced to obtain the intermediate points. The mixtures were analyzed with an accuracy of 0.1 per cent with an Orsat gas-analysis apparatus. The points fell on a straight line.

The nitrogen used was obtained from the atmosphere. That obtained from ammonium nitrite gave a different reading owing to its lack of argon. The readings obtained with this nitrogen are shown by the dotted line in figure 3. For the mixtures, oxygen was added to air in the proper proportion.

That 2 per cent of N_2 gives 30 small divisions on the drum, and as the ordinary observer can adjust the two scales to at least 5 small drum divisions, the readings may be taken with certainty to 0.3 per cent.

CALIBRATION OF INTERFEROMETER FOR OXYGEN-HYDROGEN MIXTURES.

The interferometer is even more applicable here than in the example just cited because the difference in the refractivity of the gases is greater. The difference in refractivity exceeds the limits of the scale when hydrogen is used in one chamber and oxygen in the other. With hydrogen as the standard it may be seen from figure 4 that the maximum amount of oxygen readable is about 49 per cent, as there are only 30 divisions on the scale.

With oxygen as a standard the same would be true, but it would be necessary to begin from the top of the scale and work downward, as the most refractive gas must be in the chamber *c* (fig. 1).

Figure 5 shows the apparatus used for producing pure oxygen and hydrogen by electrolysis. In bulbs *a* and *b* are platinum wires heated

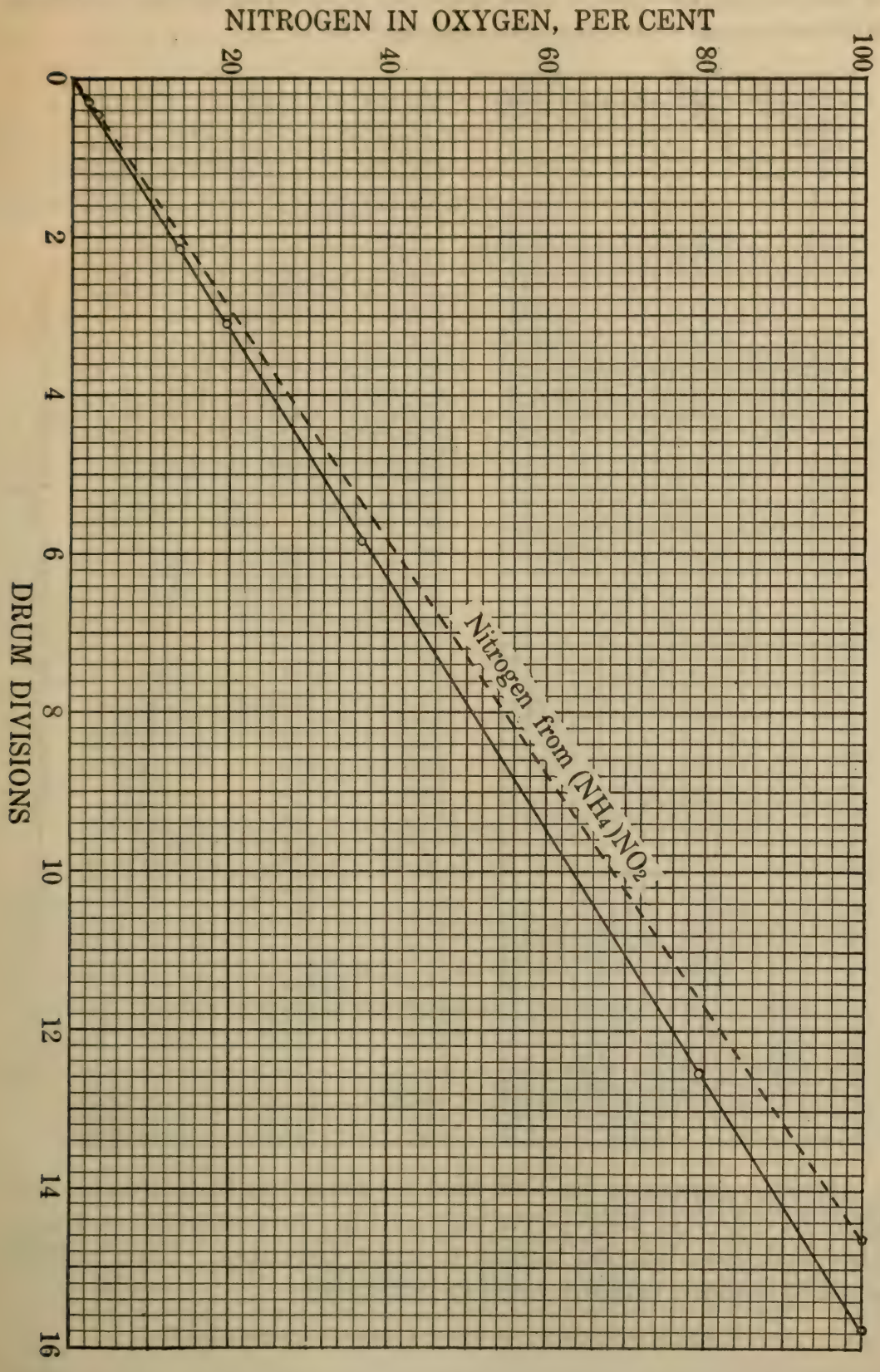


FIGURE 3.—Calibration curve for mixtures of oxygen and nitrogen.

to incandescence by an electric current. Any oxygen is removed from solution or diffusion at the positive pole and any hydrogen is re-

moved at the negative pole. The wires also break down the trace of ozone sometimes formed at the positive pole. A small wash bottle of concentrated sulphuric acid is shown at *c; d* is a calcium chloride tube and *e* a small phosphorus pentoxide tube.

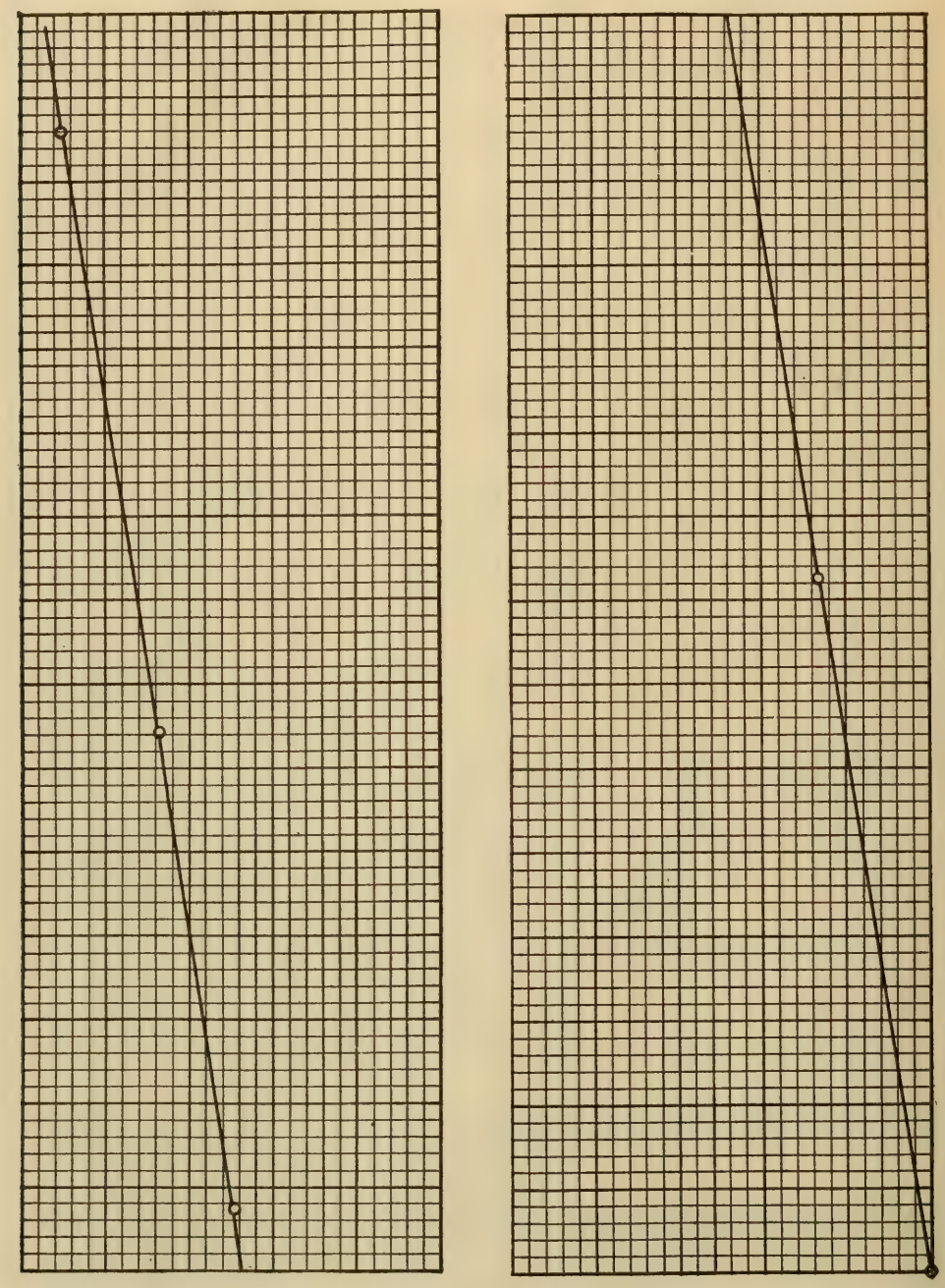


FIGURE 4.—Calibration curve for mixtures of hydrogen and oxygen.

After the apparatus has been used for some time the sulphuric acid tends to collect at the positive pole and the current becomes diminished and ozone will be formed in small quantities. Care must be taken to keep the pressure equal in both tubes.

APPLICATIONS OF THE INTERFEROMETER.

In addition to the results discussed, it seems well to outline other uses for the instrument.

As suggested by Tables 1 and 2, the interferometer may be used to investigate mine gases. The outgoing air currents from mines contain carbon dioxide and methane mixed with air. Tables 3 and 4 show that a decrease in the amount of oxygen and a corresponding increase in the amount of nitrogen are indicated by the instrument as an increase in the methane and carbon dioxide content. Hence, regardless of the cause of contamination of the air, by the addition of methane or carbon dioxide, or the withdrawal of oxygen, the instrument indicates imperfect ventilation.

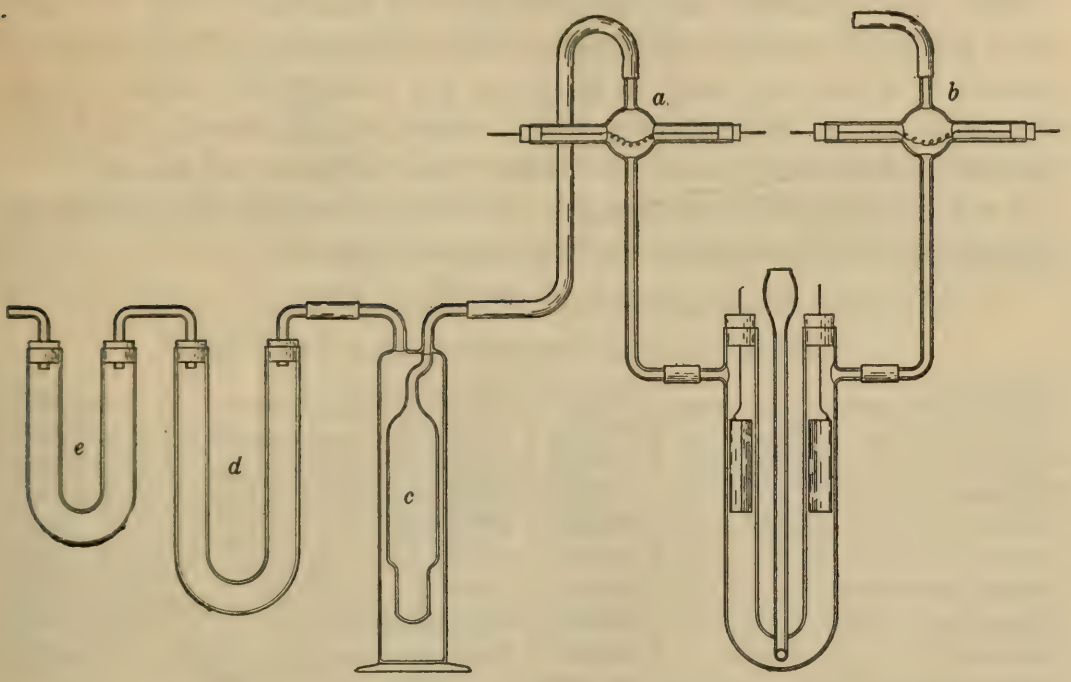


FIGURE 5.—Apparatus for preparing pure O_2 and N_2 by electrolysis.

The instrument may also be used to detect the contamination of hydrogen with heavy gases such as oxygen and nitrogen. This use should be of especial importance in determining the power of gases to carry aircraft. If air is assumed to be the contaminating gas and if pure hydrogen is used as the standard of comparison, the sensibility is 0.02 per cent. A general rule for the selection of the standard or gas of comparison is that it should be such that as the proportion of the constituent sought diminishes, the number of drum divisions of deflections will also diminish. If this relation exists, the fluctuations of the atmospheric conditions may be neglected.

A third use of the interferometer is in determining the degree of carburization of illuminating gas carburetted with benzole. The instrument should be calibrated to read in grams per cubic meter or volume per cubic foot, instead of in volumetric percentage. This may be done by passing atmospheric air through the standard chamber and by passing through the other chamber air in which a determined volume of benzole has been allowed to vaporize. A sensibility of 0.1 gram per cubic meter is to be expected.

Another application is the determination of ammonia in distillation gases. Available data show that in this determination the instrument may be made to show a sensibility of 0.3 gram per cubic meter.

A common task in gas analysis is the determination of the composition of smoke and flue gases for the purpose of boiler control. One of the chemists of the Bureau of Mines is applying the interferometer to this problem and a complete report of his results will be published by the bureau.

The interferometer may also be used to determine the efficiency of a given system of ventilation; in the control of mixtures of natural gas and air used in galleries for testing the safety of explosives; and in ascertaining the character of mixtures of sulphur dioxide and carbon dioxide resulting from organic oxidations.

As a further help in the use of the instrument the following table of the absolute indices of refraction is given.[a]

Values of absolute indices of refraction.

[0° C. and 760-mm. pressure.]

Hydrogen	1.000139	Nitrous oxide	1.000505
Water vapor	1.000261	Acetylene	1.000610
Oxygen	1.000270	Hydrogen sulphide	1.000644
Nitrogen	1.000297	Sulphurous acid	1.000665
Air	1.000293	Ethylene	1.000678
Nitric oxide	1.000300	Chlorine	1.000772
Carbon monoxide	1.000340	Cyanide	1.000834
Ammonia	1.000381	Benzole vapor	1.001700
Methane	1.000444	Pentane vapor	1.001711
Hydrogen chloride	1.000449		

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[a] Landolt, H., and Börnstein, R., Physikalisch–chemische Tabellen, 3rd ed., 1905, pp. 690–691.

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